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CONTROLLED ENVIRONMENT SOIL-CORE MICROCOSM UNIT (CESMU) FOR INVESTIGATING FATE, TRANSPORT, AND TRANSFORMATION OF CHEMICALS IN SITE-SPECIFIC SOILS

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13. ABSTRACT *(Maximum 200 words)*
The controlled environment soil-core microcosm unit (CESMU) methods embody a collection of techniques that began with soil sampling in the field, and continued throughout laboratory investigations of chemical fate, migration, and transformation in site-specific soils. Intact soil cores were collected in the field using a hydraulically-controlled probe, delivering intact soil-cores with minimal disturbance directly into high density polyethylene pipe (HDPE; 10.3-cm ID). The HDPE remained an integral part of the soil-core column. Each soil column was fitted with a porous ceramic plate in an HDPE endcap, so that a tension could be applied (30-35 kPa) to mimic field conditions, thus preventing the undue buildup of water within columns that otherwise would change soil chemical, physical, and biological properties. The intact soil-cores were under controlled temperature, and synthetic rain was added at rates simulating rainfall. Leachates were collected in darkness via Teflon tubing into flasks inside the CESMU chamber. Soil columns were harvested at intervals for sectioning by depth, extraction, and soil analyses. The CESMU methods are applicable to investigations of chemical fate, water movement, soil chemistry, solute transport/transformation, and plant effects.

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PREFACE

The work described in this report was authorized under Project No. 89PP914, Sales Order No. 1HCB. This work was started in July 1989 and completed in May 1992.

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1. INTRODUCTION

Soil column studies frequently are done to develop data on the fate and environmental effects of chemicals in terrestrial ecosystems, either as basic research or to satisfy the requirements of legislated environmental regulations (e.g. FIFRA, CERCLA, TOSCA). Too often packed soil columns are chosen for environmental studies without consideration of the impact on final results. Soil physical characteristics are typically, sometimes dramatically, altered by the drying, sieving, and storing of soil for packed columns. Such handling may also cause inappropriate and radical change in the ability of soil to degrade xenobiotics[1] or utilize naturally occurring compounds.[2] The advantages of intact soil-core columns is that they offer the potential for a more realistic view and thus assessment of soil conditions existing in the field, yet are portable so that they may be studied closely in the lab under conditions that simulate those occurring in the field. Many scientists have recognized a need for intact soil columns in soil environmental studies, and have described methods for taking soil cores via soil probes; but most of these require at least one transfer of the soil core from the probe to its destination column, potentially causing disruption of the soil core and alteration of its characteristics. However a group of scientists[3,4] have developed a soil microcosm system for taking and studying intact soil cores, and have applied their system to the extent that it was accepted as the standard method for soil microcosm research by the USEPA[5] and the ASTM.[6]

The CESMU system presented here is an adaptation of those soil microcosm methods but incorporates several important improvements, thus allowing more realistic assessment of the transport and transformation of chemicals in soil. This paper describes methods and procedures for field sampling, 1) taking and directly delivering soil cores into their respective columns with minimal disturbance of the soil sample; and for carrying out fate and effects investigations, 2) controlling the environmental parameters of the soil cores during study including soil moisture regime (matric tension; rainwater quality, quantity and rate of application), and soil temperature. These factors directly impact on the chemical, physical, and biological properties of the soil, and potentially affect the resulting transport and transformation of chemicals within soil[7] and their toxicity.[8]

2. METHODS AND MATERIALS

Materials selected were chosen primarily on the basis of their suitability for the tasks specified. Schematics shown in Figures 1-3 describe materials used, and their physical arrangement. The high density polyethylene (HDPE) pipe and endcaps used in this study were opaque, the grade and quality used in high pressure gas pipelines. HDPE pipe was purchased in 12.2-m (40-ft) lengths, then cut and sanded to the specified dimensions. Endcaps were milled to contain a well for the controlled-pore ceramic plates, then milled and threaded for tubing fittings in the endcap. Endcap fittings were HDPE.

In the field prior to sampling on-site, the soil was brought to field moisture capacity. Watering of the soil was initiated at least 24 hr before sampling to ensure sufficient time for both wetting, and drainage of excess water.

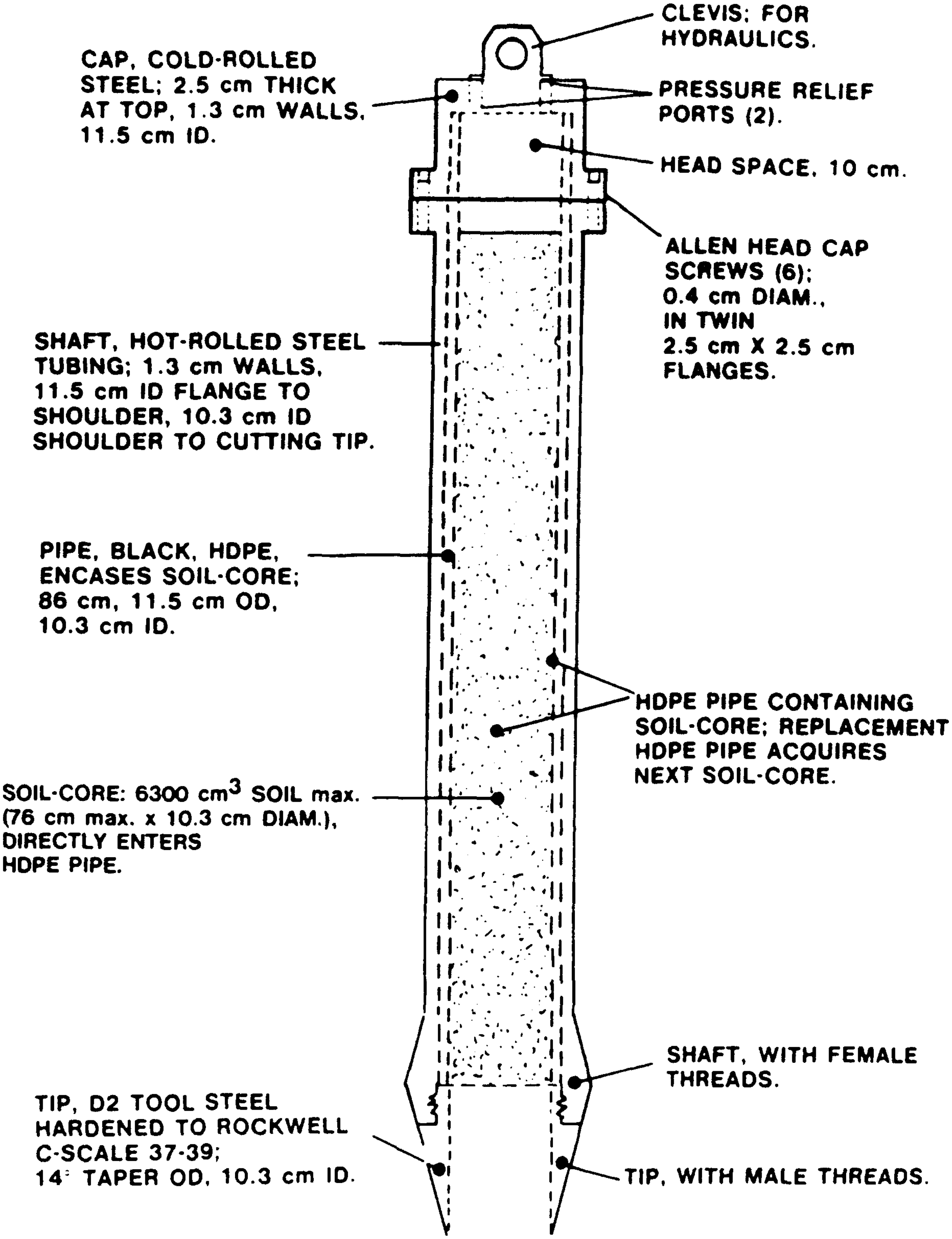

Figure 1. Cross-Section of Soil Sampling Probe with Soil Core Encased in HDPE; Detailed Materials and Dimensions.

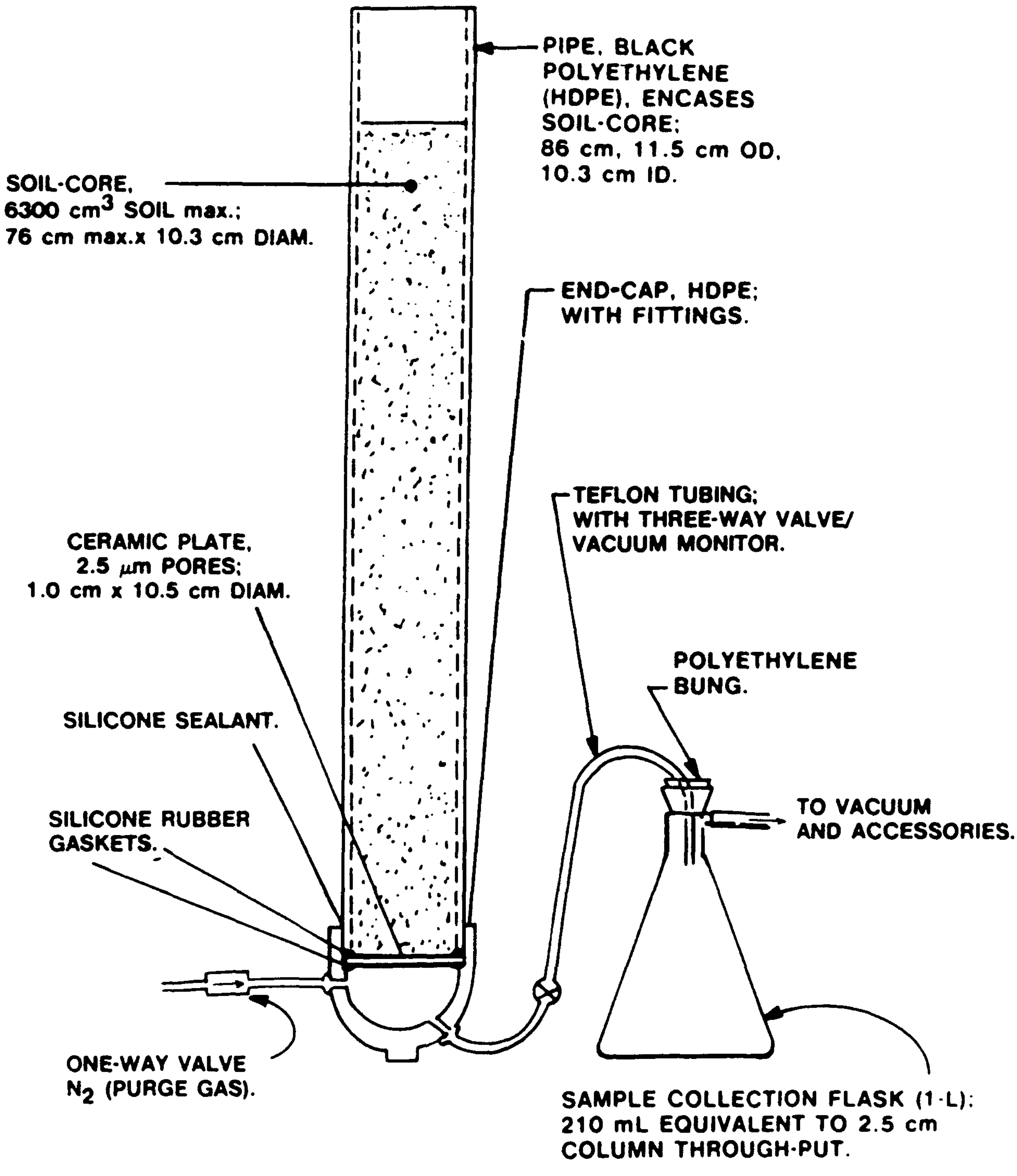

Figure 2. Diagram of Soil-Core Column Including Endcap and Fittings; Detailed Materials and Physical Arrangement.

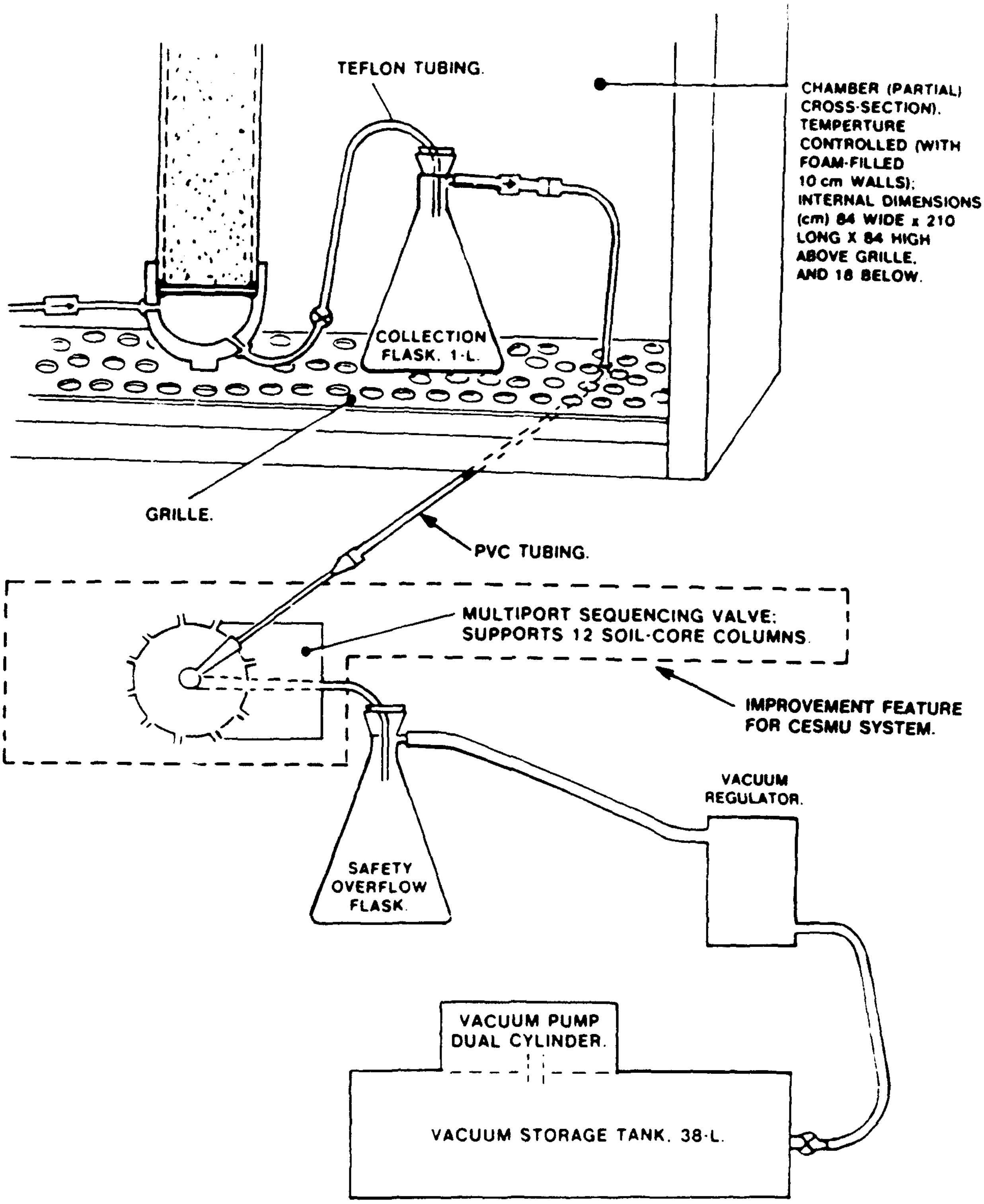

Figure 3. Diagram of Laboratory Experimental System. Details are given for one of the twelve soil-core columns per CESMU chamber. Sequencing valve and controller (shown inside dashed lines) are suggested improvements.

In the laboratory, synthetic rainwater was formulated based on records of the constituents of rainfall across Pennsylvania,[9,10,11] and used to represent the constituents and characteristics of rainfall in the mid-Atlantic coastal region. The constituents of the synthetic rainwater were (uM, in deionized water) 15 SO_4, 11 NO_3, 9 Cl, 25 NH_4, 7 Ca, 3 Mg, 1.5 Na, and 1 K; pH was adjusted to 4.6 using a 1.35:1 mixture of 1M H_2SO_4 and 1M HNO_3. This synthetic rainwater was used in simulating semiweekly rainfall events throughout the course of investigation. The amount of rainfall per event was typically determined by dividing the annual precipitation on-site by 39 weeks (9 months) and then again by 2 (semiweekly additions).

3. RESULTS AND DISCUSSION

The methods developed for use with the controlled environment soil-core microcosm unit (CESMU) controlled environmental parameters that impact on soil chemical, physical, and biological properties. The process began with soil sampling in the field, and continued throughout the laboratory investigation of chemical fate, migration, and transformation, in site-specific soils. The methodology for collecting soil cores from the field was initially developed and tested using a plot of Elkton silt loam soil (Clayey, mixed, mesic Typic Ochraquults) in Maryland. This methodology was then used successfully to collect soil cores for investigations from four sites in different states and having different soil types. These four soil types included Wheeling sandy loam [Fine-loamy, mixed mesic Ultic Hapludalfs], Lexington silt loam [Fine-silty, mixed thermic Typic Paleudalfs], Limon silty clay [Fine, montmorillonitic (calcareous), mesic Usteric Torriorthents], and Fullerton cherty loam [Clayey, kaolinitic, thermic Typic Paleudults].

The intact soil cores were collected using a hydraulically controlled probe, delivering the soil with minimal disturbance directly into high density polyethylene pipe (10.3-cm ID). Typically, a minimum of thirty columns were taken to ensure an excess of available columns from which to test and ultimately select the final twelve columns per study.[12] The soil probe was hydraulically pushed, not pounded, into the soil to alleviate zonal compaction and minimize disruption of the soil being taken.[13] By definition the act of sampling disturbed the soil, however pushing the probe into the soil (as described above) minimized detrimental effects as indicated by the measured soil bulk density (D_b) values. Soil cores from this study had D_b values that fell within the range of established values (Table 1), with relative standard deviations of only 7.5% and 2.7% for the experimental samples from the A and B horizons, respectively, of the Wheeling sandy loam.

Table. Comparison of Bulk Densities (g cm^{-3}; D_b), by Horizon, Determined for Wheeling Sandy Loam from Soil Sampled Using the CESMU Soil Probe, and the Established Range for these D_b Values

Soil Horizon	Avg. Det'd* $D_b \pm s$	Reported** D_b Range
A	1.34 ± 0.1	1.20 - 1.40
B	1.46 ± 0.04	1.30 - 1.50

* Values determined for four replicate samples.
** Soil survey of Pulaski County, VA.[14]

Native grasses at the site of sampling may also be retained within the head space above the soil sample; this can be an important factor in

studies where chemical persistence is anticipated, as individual plant species may substantially contribute to the degradation of chemicals via in situ rhizospheric microorganisms.[1,2,15] If the co-effects on/by crop species are to be evaluated, native grasses may be removed, and at the appropriate planting time individual columns may be seeded with single-species cultivars to evaluate their contribution to the degradation of chemicals of interest; the effects of crops in rotations should be evaluated individually, not be condensed into a single planting due to the potential for complex interactions among species. In these studies, native vegetation (primarily grasses) were cut at the soil surface and the aerial portions of the cut plants were removed prior to sampling the soil.

For the soil that entered the probe during collection of intact cores, the maximum clearance discrepancy allowed (using the tolerances specified, Figure 1) during delivery of soil into the HDPE pipe was <0.05-cm, with a soil-core diameter of 10.3-cm $\pm$<0.1. The HDPE pipe, an inert hydrophobic barrier, remained an integral part of the soil-core column; thus disruption of the soil due to column-to-column transfers was eliminated. Upon removal of the HDPE pipe containing the soil-core from the probe, measurements were taken of the resulting head space within each column; additionally it has been advantageous to measure the depth of soil penetration by the probe that results from sampling. If dramatic inconsistencies occurred in the depth values in the field (e.g., >20%), the corresponding columns were rejected and others taken in their place. The theoretical maximum length of the soil cores is 76-cm when the probe is inserted to its maximum depth, however reaching this limit is not normally expected under typical soil sampling conditions. Furthermore, in the field we have found it helpful to use a metal stop-plate (1-cm thick, fitted around the probe at the soil surface) so that the probe can more consistently be pushed into the soil to similar depths; such a stop-plate restricts the theoretical maximum-length of a soil core to 76-cm minus the thickness of the stop-plate selected. Thus in these studies, our theoretical maximum was 75-cm. After each sample was taken in the field, the column containing the soil core was removed from the soil probe, and each end sealed with a minimum of 8-mil thickness of polyethylene plastic to minimize gas exchange and retain soil moisture during transport to the laboratory. Afterward in the laboratory, selected soil-core columns were trimmed of excess soil if any was present, fitted with a porous ceramic disk (2.5-um pores), and opaque HDPE endcaps containing fittings for teflon tubing with in-line monitoring and shut-off valves. The intact soil columns were then transferred into the controlled temperature CESMU having a cooling capacity of 10.5 MJ h^{-1}, sufficient for maintaining a constant temperature within entire soil columns for isothermic studies at 25.0 °C, all housed in a greenhouse that provided high-temperature control. The tops of the columns were left open to receive sunlight, sufficient for plant growth; however, they can instead be covered with an opaque insulated cover spanning all columns to eliminate photo-degradative processes. (Alternatively, a top-to-bottom temperature gradient within columns can easily be created and maintained by individually elevating soil-core columns to expose the upper portion of the soil inside the column to greenhouse temperature, if thermal gradients rather than isothermal conditions are required.[16])

Controlled tension (partial vacuum) was applied equally at the bottom of each soil column across the controlled-pore ceramic plate, at 30-35 kPa; and this tension was both regulated and monitored. The tension that was applied was comparable to that encountered in the field in medium-textured soils as a result of the combined soil matric and gravitational forces.[17,18] The single improvement of adding the controlled tension to the design of the soil microcosm (especially when compared to USEPA[5] and ASTM[6] methods that rely solely upon gravitational forces), is likely the single most important improvement feature in soil microcosm design. Although applying a tension to soil columns is often done in soil science investigations, it had yet to be incorporated into soil microcosm studies.

Using this improved technique avoids undue flooding, the buildup of a hanging column of water in the lower portion of columns, and artificial changes in soil redox potential in response to steady-state alteration of the soil water content, as can happen when gravitational forces alone are relied upon to promote water flow through soil columns.

Using the improved CESMU methods the fate, migration, and transformation of nitroaromatic and nitramine explosives and their residues were investigated. However, before initiating any studies of soil chemistry, solute transport/degradation, or effects on (or by) plants, soil-core columns should be saturated with water and equilibrated under tension (48 hr minimum), after which water thru-put is evaluated for each of the initially selected columns (as was done in this study).

The selection of twelve columns per soil type (site) for initial testing was done on the basis of similarity of head space within columns, an easily obtained measurement that is the compliment to column length. Using the sampling methods described herein, the resulting column lengths typically followed a bell-curve pattern of distribution (Figure 4), resulting in a central grouping of columns differing in length by only centimeters. This grouping of columns with similar lengths provided a sufficient number of columns from which to select those for initial testing of water flow (thru-put). As expected there was a more regular and consistent frequency pattern within the groups of soil columns from the lighter-and medium-textured soils and a somewhat less regular yet satisfactory grouping pattern within the clayey and cherty (stoney) soils; only modest degradation of the frequency distribution pattern occurred even under the latter adverse soil sampling conditions.

Within each type of soil sampled, columns were initially selected on the basis of similarity of length; and replacement columns within each soil type group, if needed, were those with the next closest to the mean column length. For the initially selected columns that were found to have rates of flow or water thru-put substantially different than the median, replacement columns were selected, and then similarly evaluated. Outlier-columns within each soil type (based on values of water thru-put, when water was applied, monitored, and sampled analogous to artificial rain additions described below) were replaced until the standard deviation about the mean value for water thru-put was $\leq$10%. Then, based on the adjusted mean excluding outliers, any additional columns with thru-put values falling outside of the adjusted mean $\pm$one standard deviation were also replaced, until all test columns fell within one standard deviation of the mean. Representative columns were thus identified and retained for study in the CESMU chamber. Using these procedures to identify representative soil columns (those least affected by sampling in the field and preparation in the laboratory) has proved highly satisfactory. At this point, the chemicals selected for study was applied appropriately to the representative soil columns, and investigation of chemical fate, migration, and transformation began with the application of synthetic rainfall. If the chemical under study is best added to the study columns within a subsample of soil as was done for these, the spiked subsample should be of the same soil type as that within the study columns; soil for this purpose may be obtained from one of the unused residual columns.

The delivery and application (input) of synthetic rainwater, formulated to represent the major constituents and acidic pH of rainfall in the mid-Atlantic coastal regio.., was driven by peristaltic pumps and delivery occurred at the top of each soil-core column via capillary tubing, applied at the center of each soil-core at the rate of 7 um s^{-1} (1" hr^{-1}). Synthetic rainwater input on each column occurred twice per week on Mondays and Fridays during the course of studies, and simulated semiweekly rainfall. This method did not consider any losses of rainfall due to runoff as may occur in the field situation, and made each rainfall event equal; however, rainfall input was calculated from average native conditions at the sampling sites, and

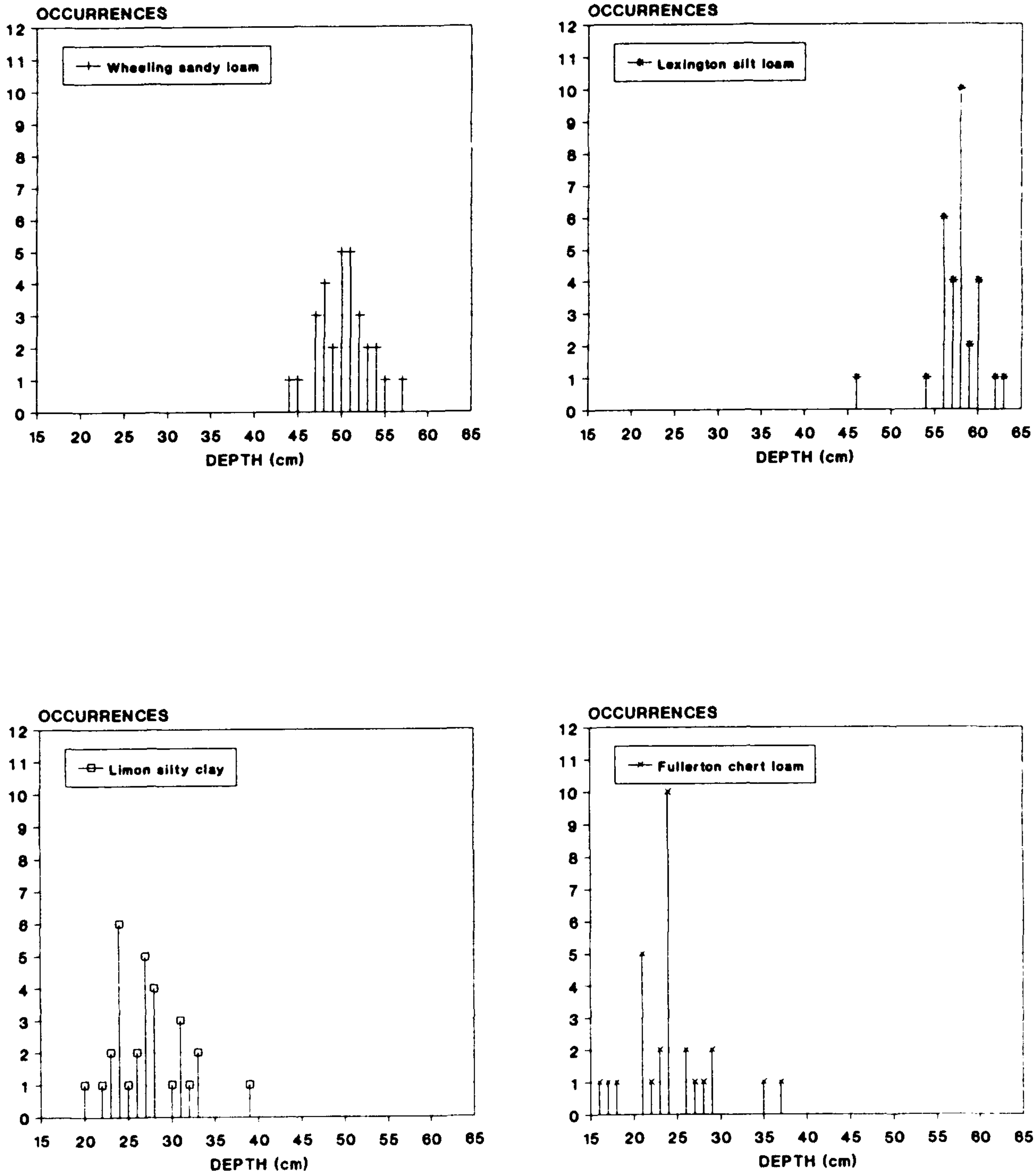

Figure 4. Frequency Distribution of Column Depth by Soil Type.

semiweekly additions allowed for wetting and drying cycles that simulated natural field conditions. Solution thru-put (leachate) was collected via teflon tubing into 1-L flasks in darkness, and kept at soil column temperature inside CESMU until removed for analyses. Nitrogen gas was provided for purging the endcap but only during collection of aqueous samples, eliminating the entry of ambient air into the bottom of columns during leachate solution removal.

Replicate treatment columns were harvested at regular intervals during the course of studies, sealed, and frozen; then the HDPE pipe containing the frozen soil-core was carefully cut length-wise using an electric router and hand guide, allowing the resulting intact soil-core to rest in the lower half of the HDPE pipe. The soil-cores were then slowly thawed in the horizontal position to effectively eliminate longitudinal migration, and the soil was sectioned into 2.54-cm (1") disks, extracted, and analyzed.

Physical and mechanical systems supporting the CESMU chamber and rainfall delivery functioned well under almost constant use for more than two years. Over this period, the fate, migration, and transformation of chemical compounds were investigated in four different soil types, using twelve study columns per soil type, with individual studies lasting from six to nine months depending upon the lability of chemicals investigated. During these studies only one study-column failed out of forty-eight total columns selected for investigation, and these remaining soil columns had relatively constant outputs within respective soil types.

Mechanical-part failures during this period included only one vacuum pump failure, replaced with a back-up unit while the original was rebuilt, and one vacuum regulator that failed inspection during an investigation and was immediately replaced with a back-up unit. Performance of the physical and mechanical systems was high, providing high confidence in maintenance of the conditions and limits designed for the studies.

4. SUGGESTIONS FOR IMPROVEMENTS

4.1 Soil Sampling.

The dimensions of the soil sampling probe described herein were designed by scaling dimensions up from a smaller less sophisticated probe that did not allow for the use of a plastic sleeve or pipe as a receptacle within the probe for the soil core. Our hydraulic probe was created *ab initio* by milling steel pipe in a metal fabrication shop; the suggestions of the metal workers were adapted regarding wall thicknesses required for physical integrity of the probe during hydraulic sampling of soil. The probe tip flared from the sample area at a rate determined from the scaling of the smaller conventional soil sampling probe; after the point at which the plastic pipe met the probe tip, the dimension of the probe receded until it equalled the external dimension of the majority of the body of the probe (Figure 1).

During the sampling of soils in the field, it was determined that drag on the probe could be substantially reduced and thereby increase sampling efficiency by reducing the effective cross-sectional area of the external flared portion of the tip and the probe. Such an increase in efficiency was obtained, when the degree of flaring was reduced. To further increase efficiency, both the length of the probe tip and the current degree of flaring were reduced. Efficiency could be even further enhanced by having the lowermost edge of the HDPE pipe that receives the soil rest within the probe tip rather than within the body of the probe; then flaring could be further reduced and perhaps totally eliminated. Testing of such a modified-probe indicated that these are viable improvements; and furthermore that the wall thickness of the probe could also be reduced to allow easier handling of the

probe in the field while retaining physical integrity of the probe and soil core during sampling.

4.2 CESMU Chamber.

Although controlled tension is applied equally at the bottom of each soil column (typically 30-35 kPa) and is regulated and monitored, the failure to maintain tension at any single column could potentially affect the tension on the remaining columns until the failing column is repaired or eliminated. Although this problem generally occurred only during the set-up and initial testing of columns and was typically a result of repairable minor leakage, it is an item of concern and a cause of extra effort that could be greatly reduced. If equal tension were applied to each column, with the tension on each column physically isolated from the remaining columns, a more secure system would result. A method for applying equal tension to isolated columns is to sequentially apply the tension to individual columns via a sequencing valve (shown inside dashed lines, Figure 3). Either a mechanically controlled or computer controlled electrical sequencing valve would allow each column to receive the same tension at intervals, without having single-column failure affect the remaining columns. Such 12-position sequencing valves are commercially available; installation and testing of computer controlled electrical sequencing valves is underway, adding this control feature option to our current CESMU systems.

4.3 Adaptability of CESMU Methodology.

CESMU methods were developed using HDPE pipe as the hydrophobic receptacle for soil cores. HDPE pipe is a common material that is relatively inexpensive yet reliable and hydrophobic, suitable for investigating the fate of many types of chemicals and materials in soil including both organics and inorganics. However, there are some organic chemicals for which metal pipe can provide better sample integrity. For these aluminum pipe is recommended, of the same dimensions as the HDPE pipe, threaded on the outside at both ends of the pipe. When materials requiring metal casings are investigated, threaded aluminum endcaps can be used in the field to secure soil cores in the aluminum column after sampling. Additional aluminum endcaps would have to be milled to accept the controlled-pore ceramic plates, and aluminum fittings for the teflon tubing. By using aluminum throughout, anomolous electrolytic reactions due to the presence of metal surfaces in contact with soil and soil leachates are minimized. Thus, chemicals incompatible with HDPE may also be investigated in intact soil cores, using aluminum pipe and endcaps.

The CESMU methods were developed so that the fate, migration, transformation and degradation of chemicals in site-specific soils could effectively be investigated, expeditiously, and in a cost effective manner. These same methods can be applied to the testing of biologically engineered organisms[19] or to testing environmental bioremediation treatments in a safe and cost-effective manner, using site-specific soils from the exact same sites anticipated for field trials. Such investigation would not only be useful in itself, but will be useful as a final screening for materials that showed promise in preliminary laboratory batch tests, prior to testing materials in the field. Screening of environmental bioremediation treatments using CESMU methods for microbiological cleanup of hydrocarbons in columns of intact soil (Udorthents) is currently underway in our laboratories. Using CESMU methodology this final screening of biotreatments is carried out in site-specific soils under site-specific environmental conditions, but under highly-controlled laboratory conditions prior to initiating high-cost field trials. By investigating the effectiveness of biological materials or treatments in this manner, only those found effective in the final CESMU screening trials need be fully investigated in the field.

5. SUMMARY

5.1 Advantages of CESMU Methods.

The CESMU methods and procedures presented herein have improved features to control parameters affecting the transport and degradation of chemicals in soil. The features that were improved (compared to USEPA[5] and ASTM[6] soil microcosm methods) include:

- Improved integrity of the physical, chemical, and biological properties of the soil sample via improved soil sampling and handling techniques

- Addition of applied tension to control the soil moisture regime

- Control of soil temperature

- Control of the quantity, quality, rate of application, and pH of synthetic rain

- Minimized alteration of redox potential of the native soil.

Disruption of native soil parameters was minimized by collecting intact soil cores using a hydraulic probe, delivering soil directly into inert hydrophobic HDPE pipe that remained an integral part of the soil-core column throughout investigations. Soil-core columns were fitted with porous ceramic plates in opaque HDPE endcaps containing fittings for teflon tubing, with in-line monitoring and shut-off valves. The intact soil columns were transferred into the CESMU chamber where controlled tension (30-35 kPa) was applied equally at the bottom of each soil column and temperature was controlled. The improvement of applying a tension (partial vacuum) to soil columns to simulate matric tension avoids undue flooding, the buildup of a hanging column of water in the lower portion of columns, and artificial changes in soil redox potential in response to steady-state alteration of the soil water content, as can happen in other microcosm systems when gravitational forces alone are relied upon to promote water flow through soil columns. Solutions were collected via teflon tubing into flasks in darkness, and kept at soil column temperature inside CESMU until harvested for analyses. Nitrogen gas was provided as purge gas during sample removal.

5.2 Adaptability of CESMU Methods.

The advantages of CESMU methodology may be applied to a wide variety of chemicals, and types of investigations, especially those affected by site-specific soil conditions. Such site-specific studies may also include investigating the effectiveness of biologically engineered organisms, or bioremediation of environmental contamination. The CESMU investigations done thusfar were cost-effective, using site-specific soils under site-specific environmental conditions in the laboratory. Even greater cost-benefit ratios could be realized if CESMU methodology were used as a screening method prior to initiating high-cost field trials involving open-release of commercial chemical products into the terrestrial environment.

6. CONCLUSION

The combined features of CESMU methodology including the improvements suggested for soil microcosm design, make this operation a state-of-the-art laboratory scale system for studying the fate and environmental effects of chemicals in terrestrial ecosystems.

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